California Environmental Protection Agency

Air Resources Board

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PROCEDURE FOR THE DETERMINATION OF DISTILLATION POINTS OF LIQUID FUELS BY AUTOMATED DISTILLATION

SOP NO. MLD 128

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SOP No. 128 - STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF DISTILLATION TEMPERATURES OF LIQUID FUELS BY AUTOMATED DISTILLATION

1 Introduction

1.1 This document describes the standard operating procedure (SOP) for measuring the distillation temperatures of liquid fuels using an automated distillation instrument.

1.2 This SOP covers gasoline, diesel fuel, aviation fuel, kerosene, and similar petroleum products.

1.3 This SOP is based on ASTM D86-90[1].

2 Method

2.1 A 100 mL sample is introduced into the instruments flask.

2.2 The sample is distilled under specific conditions depending on its characterization.

2.3 Vapor temperature and condensate volume are periodically measured. These data are used to calculate results.

3 Instrumentation

3.1 Herzog Automated Distillation unit, model MP626.

3.2 Data acquisition system: Herzog MP626 software running under Microsoft Windows 3.1

3.3 125 mL distillation flask

3.4 100 mL graduated cylinder appropriate for use with 3.1 above

4 Reagents

4.1 Toluene, A.C.S. reagent grade or better.

4.2 Mesitylene, A.C.S. reagent grade or better.

5 Preparation of Instrument

5.1 The instrument should be turned on at least 30 minutes before analyses are performed.

5.2 The correct distillation method is selected within the MP626 software. Method D86-123-1 is used for gasolines with Reid vapor pressure (RVP) greater than 9.5. Method D86-123-2 is used for gasolines with RVP less than 9.5.

5.3 The initial heater temperatures, switching time, and final heat adjustment time are selected by the operator based on the sample RVP and any previous distillation analyses performed (see 7.11).

6 Calibration

6.1 Two systems of the Herzog MP626 require calibration: temperature measurement and volume measurement.

6.2 Temperature calibration is performed by distilling pure toluene and pure mesitylene on the MP626 and comparing the result with that obtained using manual distillation with a total immersion mercury in glass thermometer. Any observed difference between the two readings is used by the MP626 software to calibrate the instrument=s temperature sensor.

6.3 Temperature calibration is carried out quarterly or whenever the temperature sensor is changed.

6.4 Volume calibration is performed by inserting an empty volumetric flask into the MP626 receiver area and selecting AZero point adjust≅ from the MP626 software.

6.5 Volume calibration is carried out quarterly or whenever the volumetric flask is changed.

7 Procedure

7.1 A piece of soft, lint-free cloth attached to a metal wire is used to clean the instrument=s condenser tube. It is inserted into the distilling compartment and pulled gently through the receiver tube.

7.2 The sample is chilled to a temperature between 32 ^{o}F and 50 ^{o}F.

7.3 The graduated cyliner and distillation flask are chilled to a temperature between 55 ^{o}F and

65 °F.

7.4 The sample is poured into the graduated cylinder. The bottom of the meniscus should line up with the 100 mL mark.

7.5 The sample is transferred as completely as possible from the graduated cylinder to the distillation flask.

7.6 The distillation flask is centered on the MP626 heating plate with the side arm connected to the inlet of the condenser tube.

7.7 The temperature sensor is inserted into the distillation flask. The sensor=s measuring element must be exactly level with the lower inside edge of the flask=s side arm.

7.8 A metallic drop plate is inserted into the top of the graduated cylinder. The cylinder is inserted under the outlet of the condenser tube. A piece of insulating material cut to fit the cylinder is used as a cover.

7.9 The distillation is started and monitored via the MP626 software.

7.10 The following parameters must be met for a distillation to be considered valid:

7.10.1 Time to first drop: 5 to 10 minutes (5 to 15 minutes for diesel)

7.10.2 Time from first drop to 5% recovered: 60 to 75 seconds (N/A for diesel)

7.10.3 Distillation rate: 4 to 5 mL/min

7.10.4 Time from final heat adjustment to end point: 3 to 5 minutes

8 Quality Control

8.1 According to ASTM D86-90, if any of the parameters in 7.10 is not met, the distillation run is not considered valid. However, in practice, failure to meet all these criteria does not make a significant difference in the data obtained. The acceptability of data obtained from an Ainvalid≅ run is determined by the client.

8.2 In order to obtain data which can meet ASTM D86-90 requirements, repeated runs with different initial temperatures, switching times, heater curve modifications, and final heat adjustment volumes are used.

8.3 Once per quarter, the distillation temperature of pure toluene is determined and compared

with a temperature determined using manual distillation with an ASTM standard mercury thermometer. Any difference between the two readings is used by the MP-626 software to correct the automated instrument=s reading.

9 References

1. "Standard Test Method for Distillation of Petroleum Products (Designation D86-90)," *Annual Book of ASTM Standards*, Vol 05.01.

2. AUser Manual for MP-626,≅ Walter Herzog GmbH, Lauda-Konigshöfen, Germany, 1995.

3. "User Manual for Software HLIS," Walter Herzog GmbH, Lauda-Konigshöfen, Germany, 1995.